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### **Report Title**

Final Report: US-Europe Workshop on Impact of Multifunctionality on Damage Evolution in Composite Materials

#### **ABSTRACT**

The US-Europe Workshop on Impact of Multifunctionality on Damage Evolution in Composite Materials was collocated with the 16th European Conference on Composite Materials (ECCM-16) (http://www.eccm16.org) in Seville, Spain. The workshop was attended by the invited presenters from the US, Europe and Australia, as well as many ECCM-16 conference participants. The presentations covered several aspects of multifunctional composites, including self-healing and self-sensing materials, the role of interfaces and multifunctional interfaces in composites, 3D experimental methods to interrogate damage in composites, integrated composite structures for energy storage and harvesting, multiscale modeling of damage, etc.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

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## **Names of Faculty Supported** PERCENT SUPPORTED NAME National Academy Member **Ioannis Chasiotis** 0.00 **FTE Equivalent:** 0.00 **Total Number:** 1 Names of Under Graduate students supported NAME PERCENT SUPPORTED **FTE Equivalent: Total Number: Student Metrics** This section only applies to graduating undergraduates supported by this agreement in this reporting period The number of undergraduates funded by this agreement who graduated during this period: ..... 0.00 The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00 The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00 Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00 Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00 The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense ..... 0.00 The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: ..... 0.00 Names of Personnel receiving masters degrees NAME **Total Number:** Names of personnel receiving PHDs **NAME Total Number:**

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## UNIVERSITY OF ILLINOIS AEROSPACE ENGINEERING



#### FINAL PERFORMANCE REPORT

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## US-Europe Workshop on Impact of Multifunctionality on Damage Evolution in Composite Materials

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#### WORKSHOP SUMMARY

The US-Europe Workshop on Impact of Multifunctionality on Damage Evolution in Composite Materials was collocated with the 16th European Conference on Composite Materials (ECCM-16) (http://www.eccm16.org) at the Hotel Barcelo Renacimiento near the University of Seville in Seville, Spain, which took place on June 22-26, 2014. The workshop was attended by the invited presenters from the US, Europe and Australia, as well as many ECCM-16 conference participants.

The workshop presentations covered several aspects of multifunctional composites, including self-healing and self-sensing materials, the role of interfaces and multifunctional interfaces in composites, 3D experimental methods to interrogate damage in composites, integrated composite structures for energy storage and harvesting, multiscale modeling of damage, etc.

The presenters reconvened in the second day of the workshop to discuss possible routes towards further development of multifunctional composites, as well as the potential scientific and technological roadblocks that need to be addressed at this stage. The discussion began with the observation that statistically significant studies are required to evaluate and further pursue multifunctional composites. Although their potential has been demonstrated, there are no data supporting the mechanical reliability of such complex systems. Therefore, a transition from proof of concept to durability studies on e.g. electrochemical performance, electromechanical coupling is due.

It was also observed that a more interdisciplinary approach (including chemists, physicists, material scientists, engineers) is needed for further advances in this field, as large part of current research on multifunctionality is led by researchers with background in traditional composites. In parallel, a new framework to evaluate the durability and performance of multifunctional materials is required, instead of evaluating and assessing each functionality independently of the rest and the overall application.

Part of the discussion focused on the current state of corporate research in the aerospace industry which is a major potential adopter of multifunctional composites. The two largest passenger aircraft manufacturers have already committed their production for the next 15 years and thus their current efforts focus in cost cutting rather than development. The supply chain also appears to be quite constrained but potential adopters of new technologies may be found among the supply companies rather than the major aircraft manufacturers. It was supported that among the currently studied composite multifunctionalities, self-sensing is the most promising and likely to be an early adopted technology. A challenging task is to combine self-sensing and self-healing.

The panel also felt that the emphasis should be placed on integrated versus added functionalities because they have the potential to result in weight reductions that the US Air Force, for instance, is interested in. In this regard, there is need for new architectures for multifunctional materials that are geared towards low volume Air Force applications. Quite later, such advanced applications may be transitioned to civilian passenger aircraft. A critical aspect of multifunctional composites is the need for repairability of both structural and of functional degradation. Towards this goal, metrics for multifunctional systems that are based on the particular application and its performance should be developed in the process of evaluating performance and performance degradation.

The panel supported that an integration of functionalities would greatly benefit from taking advantage of interfaces at the micron and the nanoscale in order to enhance multifunctionalities. Interfaces, especially between soft and hard materials and at the nanoscale have not been fully understood and well-characterized. Coordinated studies that integrate experimental and computational methods are required for further advances to be made, especially for nanostructured materials and interfaces where experiments cannot provide all the information that is needed. Currently, there is quite some understanding of the mechanical and functional role of interfaces but it is not clear how this information can be transferred between the different length and time scales. Furthermore, experimentally, speaking, there are limited tools to interrogate and evaluate the mechanical durability and performance of individual components comprising 3D multifunctional systems.

The panel concluded that current research efforts on multifunctional composites must be placed in a framework for design and manufacturability, which will support materials by design in a framework of topology optimization for multifunctional materials.

This workshop summary was compiled by Dr. Ioannis Chasiotis and edited by Dr. Edith Mäder.

Workshop Chairs: Ioannis Chasiotis (U. of Illinois at Urbana-Champaign) and Ian Sinclair (U. of Southampton)

16th European Conference on Composite Materials (ECCM-16), Seville, Spain, June 24-25, 2014 Hotel Barcelo Renacimiento, ROOM: R10-España 2

TUESDAY, JUNE 24, 2014				
Hotel Barcelo Renacimiento, ROOM: R10-España 2				
SESSION #1	Chairs: Bent F. Sørensen, (Technical University of Denmark) and Paolo Ermanni (ETH Zurich)			
9:50 – 10:00	Introductions and Opening Remarks Les Lee (AFOSR), Ioannis Chasiotis (University of Illinois Urbana-Champaign)			
10:00 - 10:20	Regenerative Polymers for Large Scale Damage Scott R. White, J.S. Moore, N.R. Sottos, Brett P. Krull, W.A. Santa Cruz, R.C.R. Gergely, *Department of Aerospace Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois, U.S.A.			
10:20 - 10:40	Continuous Self-Healing Life Cycle in Vascularized Structural Composites			
	Nancy R. Sottos, <i>Jason F. Patrick</i> , K.R. Hart, B.P. Krull, J.S. Moore, and S.R. White, *Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois, U.S.A.			
10:40 - 11:00	Multi-Functional Fibre-Polymer Composites Using Z-Pins  F. Pegorin, K. Pingkarawat, S. Daynes, <i>Adrian P. Mouritz</i> , Sir Lawrence Wackett Aerospace Centre, School of Aerospace, Mechanical & Manufacturing Engineering, RMIT University, Melbourne, Australia			
11:00 - 11:20	Modeling Strategies for Damage onset and Evolution in Composites under Complex Loading Conditions  Marino Quaresimin, Università degli Studi di Padova, Vicenza, Italy			
11:20 - 11:50	Coffee Break			

SESSION #2	Chairs: John Kieffer (University of Michigan) and Adrian Mouritz (RMIT University)
11:50 – 12:10	Material Failure of Multifunctional Composites - Key Microscale Experiments  Bent F. Sørensen, Composites and Materials Mechanics Section, Department of Wind Energy, Technical University of Denmark, Roskilde, Denmark
12:10 – 12:30	High Resolution Tomography Studies of Composites: The Data Rich Mechanics Opportunity lan Sinclair and S. Mark Spearing, University of Southampton, Southampton, UK, SO17 1BJ, UK
12:30 – 12:50	Durability and Performance of Thin Film Photovoltaics and Batteries for Integration with Composite Structures  Ioannis Chasiotis and D. Antartis, Aerospace Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois, U.S.A.
12:50 - 13:10	Damage Sensing of Carbon Nanomaterial Based Multifunctional Composites and Energy Storage Devices Tsu-Wei Chou, Ping Xu and Fancheng Meng, Center for Composite Materials and Department of Mechanical Engineering, University of Delaware, Newark, Delaware, U.S.A.
13:10 - 14:30	Lunch Break
SESSION #3	Chairs: Marino Quaresimin (Università degli Studi di Padova), Nancy Sottos (U. Illinois at Urbana- Champaign)
14:30 – 14:50	Multifunctional Nanomaterials Narayan R. Aluru, Beckman Institute for Advanced Science and Technology, University of Illinois at Urbana-Champaign, Urbana, Illinois, U.S.A.
14:50 – 15:10	Damage Evolution in Multi-Functional Composites: the Role of Interfaces John Kieffer, Mechanical Engineering, University of Michigan, Ann Arbor, Michigan, U.S.A.
15:10 – 15:30	Multifunctionality of Interphases in Composites

Edith Mäder, Department Composite Materials, Leibniz-Institut für Polymerforschung Dresden, Germany

15:30 – 15:50 **Discussion on Sessions I-III** 

WEDN	ESDAY.	<b>JUNE</b>	25, 2014
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Hotel Barcelo Renacimiento, ROOM: Andalucia 9-10

#### SESSION #4 PANEL DISCUSSION

Chairs: Ioannis Chasiotis (U. of Illinois at Urbana-Champaign), Edith Mäder (Leibniz-Institut für Polymerforschung)

10:00 - 12:00

Presenters of Sessions I-III will convene as a panel to discuss the state of the art, challenges, and future outlook of damage processes in load bearing multifunctional materials.

### **DISCUSSION TOPICS:**

- Integrated vs. added functionality: challenges and benefits (role of hierarchy of multifunctional materials, role of interfaces in multifunctionality and damage evolution, etc.)
- Metrics to evaluate and characterize (mechanical and functional) reliability and performance
- Failure processes specific to the integration of multiple functionalities in composite materials
- Design of damage tolerant multifunctional composites (e.g. bioinspired design, mechanism-based damage criteria, etc.)
- Key experimental, analytical/computational advances need to quantify, predict, and prevent material and structural failure in multifunctional, load bearing, composites.

### 12:00 – 12:20 *Closing remarks*

Les Lee (AFOSR), *Ian Sinclair* (University of Southampton), *Ioannis Chasiotis* (U. Illinois Urbana-Champaign)

## **ABSTRACTS**

#### **Regenerative Polymers for Large Scale Damage**

Scott R. White, 1,5 J.S. Moore, 2,5 N.R. Sottos, 3,5 B.P. Krull, 3,5 W.A. Santa Cruz, 2,5 R.C.R. Gergely 4,5 Department of Aerospace Engineering, Department of Chemistry, Department of Materials Science and Engineering, Department of Mechanical Science and Engineering, The Beckman Institute, University of Illinois Urbana-Champaign.

Many biological tissues can regenerate either after being damaged or as a natural consequence of the remodeling process. While self-healing has been achieved in synthetic materials, healing is limited to relatively small (microscopic) defects and cracks. In contrast, damage that involves significant mass loss like ballistic impact represents a technological challenge that has been unachievable to date. Regenerating large damage volumes is hindered by voids or large gaps in the material as liquid healing agents bleed out of the damage area since they cannot be retained there by surface tension alone. The use of scaffolds in tissue engineering circumvents this problem by providing a support network for the growth of tissue across large gaps and voids. Here we demonstrate the first synthetic system that regenerates in response to large scale damage. A two-stage polymer chemistry provides gap-filling scaffolds for shape conformity as well as robust structural performance. A vascular network delivers the reactive fluids to the site of damage. By control of reaction kinetics and delivery rate, we are able to regenerate impacted regions that exceed 35 mm in diameter. Damage-triggered regeneration will lead to synthetic materials systems capable of vastly extended life, damage tolerance, and safety.

### Continuous Self-Healing Life Cycle in Vascularized Structural Composites

Nancy R. Sottos\*, J.F. Patrick, K.R. Hart, B.P. Krull, J.S. Moore, and S.R. White \*Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, USA

Internal delamination damage in fiber-reinforced composites is difficult to detect and nearly impossible to repair by conventional methods. To date, this failure mechanism remains one of the most significant factors limiting reliability and leads to conservative design of composites for lightweight structures. In contrast to the remarkable progress in self-healing polymers, autonomous and recurrent repair of fiber-composites still presents significant challenges due to stringent processing and integration requirements. Here, we report multiple cycles of in situ delamination healing achieved through microvascular delivery of reactive fluids. Three-

dimensional vascular networks are introduced in the composite by vaporization of sacrificial fibers, with no degradation of inherent fracture properties. The vascular architecture is critical for in situ mixing, polymerization, and repeated healing of delamination damage. An interpenetrating vascular network results in full recovery (>100%) of fracture resistance after delamination, demonstrating the potential for improved safety and durability throughout the service life of high-performance composite structures.

### Multi-Functional Fibre-Polymer Composites Using Z-Pins

F. Pegorin, K. Pingkarawat, S. Daynes and Adrian P. Mouritz

Sir Lawrence Wackett Aerospace Centre, Aerospace, Mechanical & Manufacturing Engineering, RMIT University, Melbourne, Australia

This paper describes a new class of fibre-reinforced polymer composite whose multifunctional properties are controllably tailored using z-pins. Aerospace-grade carbon fibre-epoxy composite is reinforced in the through-thickness direction with thin rods (known as z-pins) to increase in combination the delamination toughness, interlaminar fatigue resistance, impact damage tolerance, and through-thickness electrical and thermal conductivities. The paper presents experimental and numerical results into the controlled modification of the multi-functional properties to carbon epoxy laminates by the judicious choice of material type, size, spatial distribution and volume content of the z-pins.

## Modeling Strategies for Damage onset and Evolution in Composites Under Complex Loading Conditions

#### Marino Quaresimin

Università degli Studi di Padova, Vicenza, Italy

In a multidirectional laminate, each off-axis ply is, in general, subjected to a multiaxial stress state leading to a matrix-dominated response and suitable criteria are needed for the initiation and propagation of intra-laminar cracks. A damage-based criterion has been developed by the author. Two parameters have been proposed as representative of the driving force for damage evolution at the microscopic scale, responsible for the initiation of ply cracks:

- the Local Hydrostatic Stress (LHS), to be used for nearly pure transverse stress conditions;
- the Local Maximum Principal Stress (LMPS), to be used when the shear stress contribution is high enough.

It has been proved that only two scatter bands, in terms of LHS and LMPS, are suitable to describe the multiaxial fatigue behavior of UD laminae, when the shear stress component is very low or high enough, respectively, compared to the transverse stress.

A procedure for the prediction of crack density evolution is proposed, based on the multiaxial criterion described above.

#### Material Failure of Multifunctional Composites - Key Microscale Experiments

#### Bent F. Sørensen

Composites and Materials Mechanics Section, Department of Wind Energy, Technical University of Denmark, Risø Campus, Building 228, Frederiksborgvej 399, DK-4000 Roskilde, Denmark

Fibre composites, consisting of long, aligned fibres in a continuous matrix material can be tailored to possess unique combinations of functionality and high mechanical strength. For instance, fibres made of shape-memory alloys enables a very large stretch and deformability. Mechanically, such behaviour can be described in terms of an in-elastic strain occurring at a certain stress level. In many cases, however, the composite behaviour may be significantly affected by process-induced residual stresses in the fibres. The presentation aims to provide some suggestions for key experiments to capture and measure such residual stresses and in-elastic strain in-situ by microscale experiments, e.g. single fibre fragmentation tests (which can be conducted under optical microscope) and in-situ observation and measurement of crack opening profiles during stable crack growth experiments in scanning electron microscope. A key issue is then to use micromechanical models to extract the in-elastic strains from the experiments.

### High Resolution Tomography Studies of Composites: The Data Rich Mechanics Opportunity

lan Sinclair and S. Mark Spearing
University of Southampton, Southampton, UK, SO17 1BJ, UK

High resolution X-ray tomography has been used to observe and quantify damage mechanisms in composite materials under load. Using synchrotron and micro-focus X-ray sources resolutions of less than 1µm have been routinely achieved. This enables individual broken fibres to be observed and crack opening and shear displacements for delaminations and intra-laminar cracks to be measured. Examples of the application of these techniques to impact damage, notch-tip splitting and fibre fracture accumulation are presented.

Quantitative data are compared to model predictions. Implications for using such high resolution 3-D measurements to inform a "data-rich mechanics" approach to materials evaluation and modeling is considered.

#### Durability and Performance of Thin Film Photovoltaics and Batteries for Integration with Composite Structures

#### loannis Chasiotis, D. Antartis

Aerospace Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois, U.S.A.

The mechanical reliability and efficiency of thin film photovoltaics attached to structural members depends on the initial state of residual stresses in the films. In this work, predictions for the mechanical and functional failure of photovoltaic films co-cured with carbon fiber composite laminates were made possible by quantifying the mean and gradient residual stresses and the failure properties of the individual layers in thin film inorganic photovoltaics. Microscale experiments provided the mean and gradient residual stresses in the micron thick photovoltaic films as well as the tensile strength and the elastic constants. The experimental results were employed to predict the onset of fragmentation of photovoltaic films that were co-cured with carbon fiber composite laminates, which were found in very good agreement with experimental measurements at the composite laminate level. Furthermore, the mechanical and electrochemical performance of composite anodes for thin film batteries were evaluated as a function of material porosity and in turn fabrication conditions. Optimal processing conditions were sought to obtained increased mechanical performance (i.e. stiffness, resilience and strength) while maintaining charge capacity. This study highlights the trade-offs in the design of loadbearing multifunctional materials where the combined performance must be evaluated.

## Damage Sensing of Carbon Nanomaterial Based Multifunctional Composites and Energy Storage Devices

### Tsu-Wei Chou, Ping Xu and Fancheng Meng

Center for Composite Materials and Department of Mechanical Engineering, University of Delaware, Newark, Delaware 19716, USA

Advancements in the science and technology of carbon nanotube (CNT) and graphene materials have provided the impetus for their application in multifunctional nanocomposites. This presentation will first review the state-of-the-art of processing, characterization and modeling of continuous CNT fiber as well as CNT/graphene hybrid fiber. Next, we explore the electromechanical behavior of

composites based on dispersed CNTs and CNT fibers, aiming for their applications in damage sensing and stretchable conductors. Thirdly, recent advancements in CNT fiber as well as graphene based energy storage devices will be reviewed. The role of processing-induced defects and material failure due to damage evolution will be examined.

## Multifunctional Nanomaterials Narayan R. Aluru

Beckman Institute for Advanced Science and Technology, University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA

Emerging nanomaterials offer exciting possibilities for multifunctional behavior – for example, nanomaterials can be used to probe mechanical, electrical, chemical, and other physical phenomena. Some of the strongest nanomaterials that have been reported to date include materials such as graphene, carbon nanotubes, bucky balls, etc. In this talk, we will discuss the unique multifunctional behavior of nanomaterials. First, we will discuss the strength and failure of graphene. The exceptional mechanical properties of graphene make it a promising nano-resonator to detect tiny masses approaching the femtoscale and smaller. However, the intrinsic losses in nanomaterials can limit their performance. We will discuss the intrinsic losses and present a multiscale approach. Second, graphene-based hetero-structures (e.g. graphene/SiO2 composite) are promising candidates for chemical sensing. Specifically, we show that engineering defects into nanomaterials can provide unique electronic behavior, thereby enhancing the sensitivity of nanomaterial composites. Third, bucky-ball-based polymer composites are promising multifunctional materials. We will discuss the strength and failure of bare and water-filled bucky-balls and discuss the significance of quantum phenomena in failure of nanomaterials. Finally, if time permits, we will discuss the structure and physical properties of self-assembled biomolecular multifunctional nanostructures.

### Damage Evolution in Multi-Functional Composites: the Role of Interfaces

#### John Kieffer

University of Michigan, Ann Arbor, MI 48109, U.S.A.

Multi-functionality in materials is often achieved by combining the functionality of different components through forming composites. This naturally creates interfaces and interphases in these materials, which can become the sites for damage initiation and

propagation. To mitigate damage evolution, we need to better understand the structure and properties of interfaces, especially between dissimilar materials, and the adjacent regions. However, study of interfaces is encumbered by the fact that they are typically buried, thus difficult to access, and of small spatial extent perpendicular to their defining plane. In this presentation we provide some examples of recent advances in the characterization of interfaces in materials combining organic and inorganic phases. The methods of investigation strongly rely on the intimate coupling between experiment and simulations. We present some new results based on inelastic light scattering, thermal transport, and secondary ion mass spectrometry measurements, in conjunction with atomistic simulations. We discuss the insights gained from these studies. The presentation will be broad and multifaceted with the intention to stimulate discussion of novel materials design approaches towards multi-functional composites with improved damage monitoring and damage control capabilities.

#### **Multifunctionality of Interphases in Composites**

#### Edith Mäder

Department Composite Materials, Leibniz-Institut für Polymerforschung Dresden, Germany Institute of Materials Science, Technische Universität Dresden, Germany

Nanoscale materials (CNTs, GNPs) have been employed into composites' interphase in order to implement multiple functions, such as mechanical, electrical, hydrothermal and chemical. They were deposited onto electrically insulating glass fibre surfaces, leading to the formation of semiconducting CNTs-glass fibres and in turn multifunctional fibre/polymer interphases. First, the CNTs-glass fibre demonstrated a high sensitivity towards strains by simultaneous determination of electrical resistance under tensile loading, which highlights a potential as in-situ micro sensor in materials. Secondly, the interfacial shear strength of single fibre composites exhibited more than 30 % improvement. Third, an in-situ strain sensor was manufactured to real-time monitor the microcrack in composites instead of external sensors. Finally, the CNTs-glass fibre was used as an in-situ physical-chemical sensor to characterize the processes of curing or crystallization of polymers through monitoring the low mobility of charge carriers in insulative materials. The glass transition was interpreted by the resistance measurement.